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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification 5 :</b> <b>C02F 1/52, 9/00, D21C 5/02</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 93/02967</b> <b>(43) International Publication Date:</b> 18 February 1993 (18.02.93)
<b>(21) International Application Number:</b> PCT/GB92/01430 <b>(22) International Filing Date:</b> 3 August 1992 (03.08.92)  <b>(30) Priority data:</b> 9116701.5                      2 August 1991 (02.08.91)                      GB  <b>(71) Applicant (for all designated States except US):</b> ALLIED COLLOIDS LIMITED [GB/GB]; P.O. Box 38, Low Moor, Bradford, West Yorkshire BD12 0JZ (GB).  <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only) :</b> LANGLEY, John, Graham [GB/GB]; Thorpe Lane, Guiseley, Leeds, West Yorkshire LS20 8NS (GB). FORD, Philip, Anson [GB/US]; 8957 River Crescent, Suffolk, VA 23433 (US). TREMONT, Stephen, Robert [US/US]; 3201 St. Anthony, Portage, MI 49002 (US). SATTERFIELD, Brian, Frederic [US/US]; 1405 Brookview Road, Columbia, SC 29212 (US).		<b>(74) Agent:</b> LAWRENCE, Peter, Robin, Broughton; Gill Jennings & Every, 53 to 64 Chancery Lane, London WC2A 1HN (GB).  <b>(81) Designated States:</b> AT, AU, BB, BG, BR, CA, CH, CS, DE, DK, ES, FI, GB, HU, JP, KP, KR, LK, LU, MG, MN, MW, NL, NO, PL, RO, RU, SD, SE, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, SN, TD, TG).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> DEWATERING OF AQUEOUS SUSPENSIONS  <b>(57) Abstract</b>  A suspension which is preferably an ink-containing waste suspension recovered from a paper deinking process is flocculated by a cationic polymeric flocculant, and the flocculated material is then aggregated by an anionic colloidal material mixed into the flocculated suspension, optionally further cationic flocculant is added, and the aggregated material is then separated from the suspension by belt press or other pressure filtration.		

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### Dewatering of Aqueous Suspensions

This invention relates to a process for separating suspended solids from an aqueous suspension and which includes a dewatering stage that is conducted by pressure filtration.

The invention is of particular applicability to processes in which the suspension contains hydrophobic material such as ink particles, and especially when the suspension is a suspension of waste material in a paper deinking process.

It is well known to add a flocculant material to a suspension so as to cause the suspended material to flocculate before dewatering. Suitable flocculants for this purpose include alum and other polyvalent metal salts, bentonite, and various natural or synthetic polymers that can be non-ionic, anionic or cationic. In some instances combinations are known, for instance bentonite followed by a polymer. Some of the flocculant materials might be considered, more accurately, to be coagulants rather than flocculants.

The choice of flocculant is dictated by the particular process steps that are involved, the materials that are being flocculated, and the economics of the process. For instance a process that is designed to make a product that can be sold may justify a more expensive flocculant than a process designed to make a waste product. Similarly, a process designed to flocculate essentially hydrophilic materials will generally require different flocculants from a process designed to flocculate essentially hydrophobic materials.

A flocculation process useful in the production of paper is commercialised under the trade name "Hydrocol" and is described in EP-A-235893. In this a substantially linear synthetic cationic polymer having molecular weight above 500,000 is added to a cellulosic suspension to form flocs which are then broken down by shearing to form

microflocs, and bentonite is then added to the sheared suspension, which is then drained through a screen to form a paper sheet. This sheet can then be dried over hot rolls or in an oven. It is essential in this process to  
5 apply sufficient shear to break the initial flocs down to microflocs as otherwise the paper that is formed by drainage through the screen will have bad formation and there will be inefficient drying of the paper in the oven or over the hot rolls because moisture will be trapped in  
10 the flocs.

The process is clearly of no relevance to a process where the dewatering is by a different technique (pressure filtration) the suspended solids are entirely different (generally containing hydrophobic ink) and the intended  
15 product is a cake that will frequently be dumped, instead of being a sheet of paper that can be sold.

In order to dewater by pressure filtration a suspension containing hydrophobic particles such as the insoluble ink particles obtained in a paper deinking  
20 process it is common to add a flocculant before the pressure filtration, and the flocculant is normally a high molecular weight (for instance intrinsic viscosity above 4dl/g) cationic polymer.

It would be desirable to be able to improve the  
25 process, by using a more effective flocculation system, in order that the cake obtained by pressure filtration could be obtained more easily and, in particular, to a higher dry matter content.

A process according to the invention for separating  
30 suspended solids from an aqueous suspension comprises flocculating the suspended material by mixing a cationic polymeric flocculant into the suspension, aggregating the flocculated material by mixing an anionic colloidal material into the flocculated suspension, and separating  
35 the aggregated material from the suspension by pressure filtration.

The pressure filtration can be effected by centrifugal dewatering (since the centrifugal force will have the effect of applying pressure) or by any other conventional pressure filtration system such as a filter press, screw  
5 press, belt press or tube press.

The process can be used to collect a cake that is industrially useful (for instance a pigment cake) that can then be broken and used in a desired manner, but it is often preferred to apply the invention to the separation of  
10 waste solids from a liquor, in which event the cake obtained by pressure filtration may be dumped.

The invention has the advantage that it permits the dewatering stage to be conducted very rapidly and very efficiently and it can be performed to give a cake having  
15 a higher solids content than is often obtained by conventional processes.

The suspension that is to be dewatered can be a suspension of organic or inorganic particles, and the particles may initially be colloidal or dispersed.  
20 Depending on the nature of the particles, the flocculant may either act as a conventional bridging flocculant or it may act more as a coagulant than as a flocculant, and in some instances it is desirable to add one polymeric material that will act as a coagulant and another that will  
25 act as a bridging flocculant.

The suspension can be a suspension of sewage solids, for instance a sewage sludge or raw sewage, or it can be an industrial effluent or an industrial liquor that is being used to recover waste material. For instance the  
30 suspension can be an effluent that is to be discharged after dewatering or it can be a suspension that is to be recycled after removing most of its suspended solids, for instance for dumping. Alternatively, the suspension can be a suspension of industrially useful material, for  
35 instance a pigment suspension from which pigment is to be separated.

The invention is of particular value when the suspension comprises hydrophobic material, and in particular when the suspension is a suspension containing at least 5% by weight (based on total solids) insoluble ink particles. Such a suspension is obtained in a paper deinking process and thus the process of the invention is preferably incorporated as part of a paper deinking process and results in the production of a cake of waste material that may then be dumped. In particular, the suspension is preferably the hydrophobic reject suspension obtained in a paper deinking process by subjecting a suspension of paper fibres, filler and ink to flotation and thereby separating a hydrophobic reject suspension from a hydrophilic accept fraction. Frequently the hydrophobic reject suspension is a blend of suspensions collected from two or more flotation stages in the overall deinking process. Such a process is illustrated diagrammatically in the accompanying flow diagram.

Cellulosic material that is contaminated with ink (e.g., printed newsprint or printed cardboard) is subjected to a pulping and deinking process that generally involves a series of steps including various bleaching and screening steps (that are not shown) to form an aqueous pulp that is passed along line 1 to a dewatering stage 2. This dewatering stage is generally an air flotation stage in which the ink-containing solids are floated off as a Reject slurry, that is removed along line 3 to a collection vessel 4, while the hydrophilic Accept liquor is passed through line 5 to one or more stages 6 in which the liquor is concentrated (e.g., by drum or other filtration) and the solids in it are washed and filtered. The useful solids from these stages are taken through line 7 to a stock tower for recycling while ink-contaminated wash water is taken by line 8 towards a Krofta clarifier or other flotation cell 9. The hydrophobic Reject slurry that is floated in this cell is taken by line 10 to the vessel 4, while water can be removed by line 11 and recycled.

Chemicals to promote the flotation can be added at the dosing system 12.

In addition to feeding the reject slurries from flotation cells 2 and 9 to the vessel 4, frequently other reject solids, for instance general mill effluent solids, may be fed to it through line 13.

The slurry accumulated in vessel 4 is passed through line 14 and dosing system 15 to an Adritz or other belt press 16 where it is subjected to pressure filtration to produce a water effluent removed through pipe 17 and a cake that can be removed, for instance as shown at 18, and can then be dumped, for instance as landfill.

The slurry that is to be filtered typically has a solids content of below 5%, usually below 3% and often below 1%, for instance below 0.5%. The solids content is usually at least 0.05%, and is often at least 0.1%. The solids content of this slurry typically contains 10 to 70% (often 20 to 40%) by weight cellulosic fibres and/or fines, 0 to 80% (often 30 to 70%) pigment and other filler, and 5 to 50% (often 10 to 30%) insoluble ink. The ink may have initially been water insoluble or may have been insolubilised during the process.

The flotation stage or stages may be conducted as dispersed air flotation processes in which air at atmospheric pressure is forced into the liquor, or they may be conducted as dissolved air flotation processes in which the in-flowing liquor is pressurised in the presence of air so as to dissolve air in the liquor, and the pressure is released in the flotation cell (or alternatively the inflowing liquor may be under atmospheric pressure and the flotation cell may be under reduced pressure). Irrespective of the precise manner of effective air flotation, the central feature is that solids are floated to form a Reject fraction, and the clarified liquor remains as the Accept fraction.

The use of a belt press is particularly preferred.

The cationic polymer that is used in the invention can be a natural cationic polymer such as chitosan or a modified natural cationic polymer such as cationic starch. Preferably however the polymer is an organic synthetic polymer that is substantially water soluble and that is formed by polymerising one or more ethylenically unsaturated monomers, in generally acrylic monomers, that consist of or include cationic monomer. Suitable cationic monomers are dialkylaminoalkyl (meth) acrylates and dialkylaminoalkyl (meth) acrylamides, either as acid salts or preferably as quaternary ammonium salts. The alkyl groups may each contain 1 to 4 carbon atoms and the aminoalkyl group may contain 1 to 8 carbon atoms. Particularly preferred are dialkylaminoethyl (meth) acrylates, dialkylaminoethyl (meth) acrylamides and dialkylaminopropyl (meth) acrylamides. These cationic monomers are preferably copolymerised with a non-ionic monomer, preferably acrylamide. Cationic amphoteric polymers (including a minor amount of anionic groups) can also be used. Preferred polymers are particulate as in EP 202780.

Various other cationic polymers that may be used include polyethylene imines, dicyandiamide polymers, polyamine epichlorhydrin polymers and polymers of diallyl monomers such as diallyl methyl ammonium chloride, either as homopolymer or copolymer with acrylamide or other comonomer.

The polymer can have sufficiently high molecular weight (e.g., intrinsic viscosity above 4dl/g) that it is a bridging flocculant. However it is often preferred that the cationic polymer that is added to the suspension has a sufficiently low molecular weight that the flocculation process can more properly be described as coagulation. Preferably the molecular weight of the polymer is such that IV is not above 3dl/g, e.g., 0.2 to 3dl/g or molecular weight 50,000 to 2 million. Suitable low molecular weight polymers of this type are low molecular weight versions of



polymers of one or more ethylenically unsaturated monomers including cationic monomer, as discussed above, and the polyethyleneimine dicyandiamide, polyamine epichlorhydrin polymers, and polymers of diallyl monomers, as discussed  
5 above.

The cationic polymer generally has a relatively high charge density, for instance above 0.2 and preferably 0.4 to 2.5 equivalents of cationic nitrogen per kilogram of polymer. When the cationic polymer has IV below about  
10 3dl/g the cationic content is preferably relatively high. For instance the polymer may be a substantial homopolymer or formed from a monomer blend at least 50% and generally at least 80%, by weight cationic monomer, any remaining monomer generally being acrylamide or other non-ionic  
15 monomer. At higher molecular weight it can be satisfactory for the amount of cationic monomer to be, for instance, 8 to 40, often around 10 to 20, mole percent.

In some instances it is desirable to include a relatively low molecular weight cationic polymer (for  
20 instance a relatively highly charged cationic polymer having molecular weight 50,000 to 2 million) as a coagulant followed by a higher molecular weight cationic polymer, for instance to act as a bridging flocculant, followed by the anionic colloidal material. For instance the coagulant  
25 can remain from an earlier stage in the process and the flocculant can be added to the slurry prior to pressure filtration.

Although the process can be conducted with relatively low degrees of mixing, with the result that there is little  
30 or no degradation of the initial flocs before the anionic colloidal material is added, it is often preferred to break the flocs down into smaller flocs before adding the anionic colloidal material. This reduction in floc size can be  
35 achieved by applying stirring or other agitation to the flocculated suspension. Naturally the extent of agitation must not be so great that the initial solids are resuspended in the aqueous suspension but it is possible,

particularly if relatively large amounts of the cationic polymer flocculant are used, to choose a degree of agitation that breaks the initial flocs down to microflocs that are stable in the system against further reduction in size, and which are then aggregated by the anionic colloidal material. An advantage of reducing the floc size in this manner is that it can facilitate dewatering to a higher solids content compared to the solids content than is conveniently available if the flocs do not undergo size reduction before adding the anionic colloidal material. Size reduction of the flocs by agitation is particularly desirable when the polymer has IV above 4dl/g.

The addition of cationic polymer is made at some position ahead of the belt press or other pressure filtration apparatus and anionic colloidal material is added after the polymer addition but before the pressure filtration stage. There must be sufficient interval between the two points of addition to allow flocculation to occur and, as indicated above, it can be desirable to apply sufficient mixing or other shear between the two points of addition so as to break down the initial flocs that are formed into microflocs.

The anionic colloidal material can be an emulsion of water-insoluble anionic organic polymer, or it can be an anionic inorganic polymer such as colloidal silicic acid, but preferably it is an anionic swelling clay. Such clays are known to swell to a very large extent when the dry clay is contacted with water. They are generally smectites and are frequently montmorillonites. Suitable materials are referred to as bentonites such as Wyoming bentonite, or Fullers Earth. The bentonite or other clay may have been chemically modified, e.g., by alkali treatment to convert calcium bentonite to alkali metal bentonite. The bentonite or other clay preferably swells by at least 10 or 20 times its own volume and the dry clay is contacted with water and the surface area of the anionic colloid before swelling is preferably at least  $30\text{m}^2/\text{g}$  and the surface

after swelling is preferably at least 400, e.g, up to 800m<sup>2</sup>/g.

The bentonite or other clay may be provided as a powder that is mixed with water to form a slurry for convenient addition to the aqueous suspension, or may initially be provided as a slurry, and this in turn may be provided as a concentrated slurry that contains low molecular weight sodium polyacrylate or other dispersing agent or other material that will render the concentrate sufficiently fluid for ease of handling.

The amounts of cationic polymeric flocculant and anionic colloidal material used in the invention will depend upon the suspension that is being treated and the degree of agitation that is applied and the nature of the pressure dewatering process. The optimum can be determined by routine screening. If the flocculated suspension is to be agitated significantly prior to adding the anionic colloidal material, it is desirable to include sufficient cationic polymer to ensure that the microflocs that are present after the agitation all carry a relatively heavy cationic charge due to the flocculant polymer. The amount of polymer that is included in the suspension is generally at least 2ppm (based on the weight of suspension) and is generally at least 5 or 10ppm. It can be up to, for instance, 500ppm but the amount is generally below 100ppm and is frequently below 50ppm. Values of 5 to 50, often around 20 to 30, ppm are often preferred. These are all based on the total weight of suspension. Based on the solids content of the suspension, the amounts typically are at least 0.1% and often at least 0.5%, but generally below 5% and often below 2%, by weight of the solids content of the dispersion.

The amount needed for optimum results may be reduced if the suspension already contains cationic polymer from a previous stage, for instance from a previous flotation stage.

The amount of anionic colloidal material is generally at least 10ppm and usually at least 50ppm and preferably at least 100ppm, based on the weight of the dispersion. The amount is generally below 500ppm and frequently it is below 250ppm. Amounts around 50 to 150ppm are often preferred. These amounts are based on the weight of the aqueous suspension that is to be subjected to the pressure filtration. Based on the solids content of the suspension, the amounts typically are above 0.1% and generally above 0.3% and often above 1%, but the amount is generally below 5%, and preferably below 3% by weight.

In addition to providing the cationic polymeric flocculant in the suspension and then aggregating it by adding anionic colloidal material prior to pressure filtration, it can be desirable to add further cationic polymeric flocculant to the suspension after adding the anionic colloidal material and before the pressure filtration. This added cationic material is generally of high molecular weight (for instance intrinsic viscosity above 4dl/g at 25°C in 1N sodium chloride solution) and can have relatively low charge density, for instance being formed from 3 to 25 mole percent, often 8 to 15 mole percent, cationic monomer with the balance acrylamide.

It is generally preferred that the solids in the resultant cake are used or dumped without further drying (for instance by heating or combustion) but if desired they can be subjected to further treatment before use or dumping.

As an example of the invention, waste inked paper is pulped in the presence of alkali silicate and chelating agent, bleached and filtered and soap is then added to the suspension.

Referring to the drawing, the suspension is then subjected to air flotation at 2 to form a first Reject slurry (line 3) and an Accept liquor which is thickened and washed at 6 by suction drainage or passage over a drum thickener or screw press, and the filtrate is taken to a

clarifier 9 while the solids are rewashed and again filtered, with the washings being taken to the clarifier.

This clarifier 9 is a Krofta flotation cell in which the liquor is subjected to air flotation with the clarified  
5 Accept liquor being recycled to the pulping stage and a second Reject slurry being collected.

The first and second Reject slurries are mixed together at 4 to form a slurry having a solids content of which about 50% is filler, 30% fibre fines and 20% ink.  
10 The addition of the second Reject is desirable as it increases the fibre content of the combined slurry and so improves its handling.

In the process of the invention, two-stage dosing is conducted at the dosing system 15, with 25ppm homopolymer  
15 of diallyldimethyl ammonium chloride having IV 2dl/g being mixed into the slurry followed by 100ppm bentonite.

In a comparative process (not in accordance with the invention) the two-stage dosing consists of adding the bentonite followed by a conventional high molecular weight  
20 cationic polymer.

Preferably a cationic polymer of 10 mole % quaternised dimethylaminoethyl acrylate and 90 mole % acrylamide and having IV above 8dl/g is added at a dosage of 100g/t after the addition of bentonite and before the pressing.

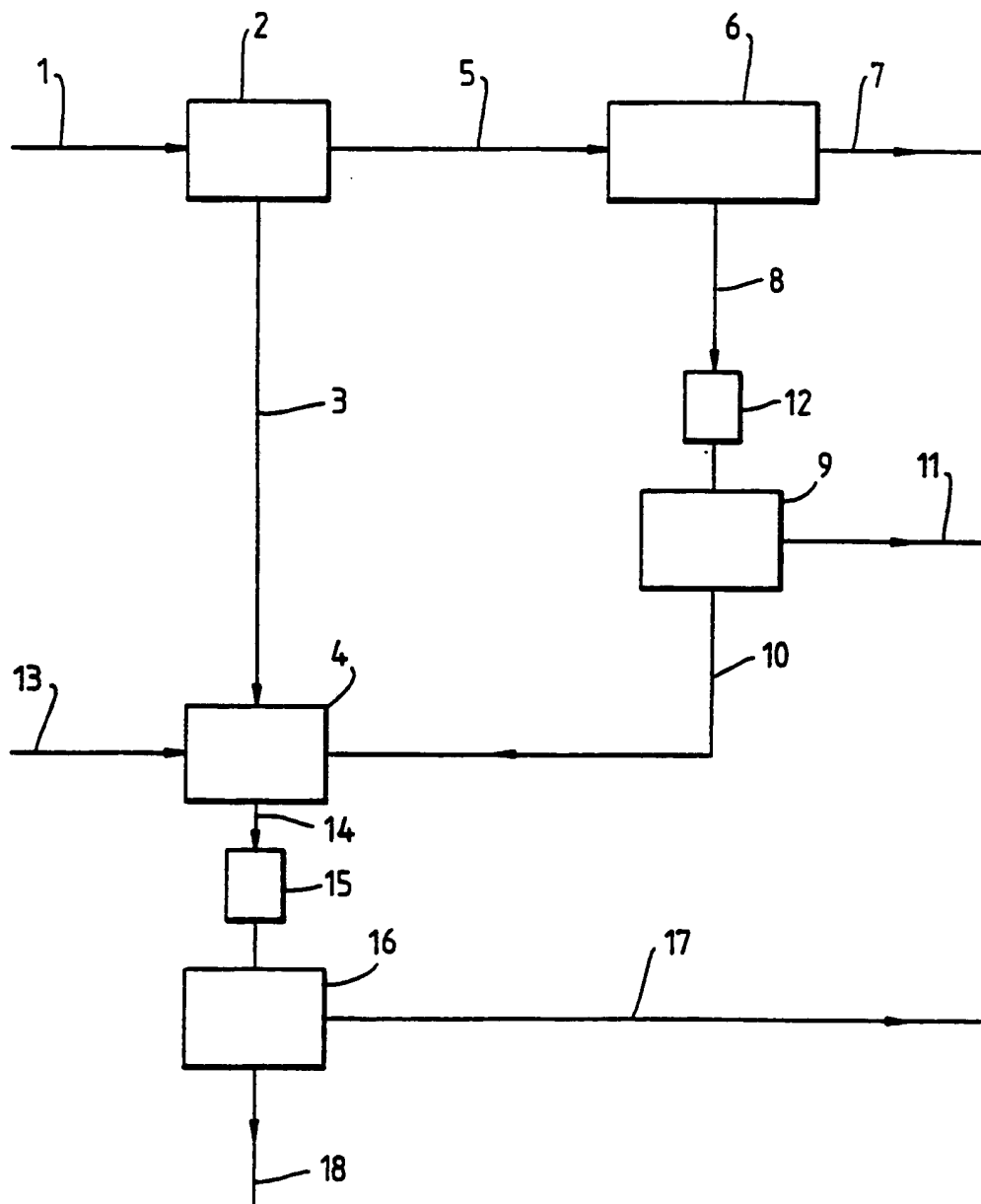
25 The process of the invention in this example is capable of giving a filter cake having a solids content of 38%, compared to a solids content of around 32% when conducted by the prior process.

CLAIMS

1. A process for separating solids from an aqueous suspension by mixing a cationic polymeric flocculant into the suspension and then separating the solids from the suspension by pressure filtration to form a cake, characterised in that the flocculated solids are aggregated by mixing an anionic colloidal material into the flocculated suspension and the suspension containing the aggregated solids is subjected to the pressure filtration.
2. A process according to claim 1 in which the suspension is a suspension containing at least 5% by weight (based on total solids) insoluble ink particles.
3. A process according to claim 2 in which the suspension is a suspension containing 5 to 50% by weight insoluble ink particles, 10 to 70% by weight cellulosic fibres and fines, and 0 to 80% by weight filler, the percentages being based on total solids.
4. A process according to claim 2 or claim 3 in which the suspension is the Reject suspension obtained in a paper deinking process by subjecting a suspension of paper fibres, filler and ink particles to flotation and thereby separating a hydrophobic Reject suspension from a hydrophilic Accept fraction.
5. A process according to any preceding claim that is part of a paper deinking process, and in which the cake is dumped.
6. A process according to any preceding claim in which the pressure filtration is by a belt press.
7. A process according to any preceding claim in which the cationic polymer is a water soluble organic synthetic polymer selected from polymers of cationic ethylenically unsaturated monomer, polyethylenimine, dicyandiamide polymers, and polyamine epichlorhydrin polymers.
8. A process according to any preceding claim in which the cationic polymer comprises polymer that has IV 0.2 to 3dl/g and is formed from monomer of which at least 50% by weight is cationic.

9. A process according to claim 7 in which the cationic polymer comprises polymer having IV at least 4dl/g.
10. A process according to any preceding claim in which the flocculated solids are broken down to microflocs by stirring before adding the anionic colloidal material.
- 5 11. A process according to any preceding claim in which the suspension containing the aggregated solids is flocculated by adding a second cationic polymeric flocculant before the pressure filtration, wherein this
- 10 12. A process according to any preceding claim in which the anionic colloidal material is an anionic swelling clay.

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## INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 92/01430

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (If several classification symbols apply, indicate all) <sup>6</sup>		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl. 5 C02F1/52; C02F9/00; D21C5/02		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched <sup>7</sup>		
Classification System	Classification Symbols	
Int.Cl. 5	C02F ; D21C	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched <sup>8</sup>		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT<sup>9</sup></b>		
Category <sup>10</sup>	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
Y	EP,A,0 406 105 (LA CELLULOSE DU PIN) 2 January 1991 see the whole document ---	1-5
Y	US,A,3 617 568 (RIES,H.E.JR) 2 November 1971 see the whole document ---	1-5
A,P	US,A,5 071 587 (PERMAN,C.A.) 10 December 1991 see claims 1-4,21 ---	1,7,12
A	GB,A,867 450 (INDUSTRIAL RAYON CORPORATION) 10 May 1961 see page 2, line 90 - line 121 ---	1,12
-/--		
<p><sup>10</sup> Special categories of cited documents : <sup>10</sup></p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&amp;" document member of the same patent family</p>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
13 NOVEMBER 1992	23. 12. 92	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	GONZALEZ ARIAS, M.L.	

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	FR,A,1 472 923 (THE HOLLIFIELD CORPORATION) 17 March 1967 see the whole document -----	1-5

**ANNEX TO THE INTERNATIONAL SEARCH REPORT  
ON INTERNATIONAL PATENT APPLICATION NO. GB 9201430  
SA 62919**

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on  
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A-0406105	02-01-91	FR-A- 2649091	04-01-91
		CA-A- 2020069	30-12-90
		JP-A- 3131384	04-06-91
US-A-3617568	02-11-71	None	
US-A-5071587	10-12-91	WO-A- 9118838	12-12-91
GB-A-867450		US-A- 3046233	
FR-A-1472923		DE-A- 1517666	01-10-70
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